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CLAIMS

(57) [Claim(s)]

[Claim 1] an empirical formula -- $100(\text{Fe}_{1-m}\text{Tm})_x\text{-y-zQxRyMz}$ (one or more sorts of elements chosen from the group which T becomes from Co and nickel --) One or more sorts of elements chosen from the group which Q becomes from B and C, one or more sorts of rare earth metal elements with which R does not contain La and Ce substantially, M is the metallic element chosen from the group which consists of Ti, Zr, and Hf. It is expressed by at least one sort of metallic elements which surely contain Ti. The presentation ratio x, y, z, and m, respectively $x < 10 < 17$ atom %, $6 \leq y \leq 9.3$ atom %, Satisfy $0.1 \leq z \leq 6$ atom % and $0 \leq m \leq 0.5$, and two or more kinds of ferromagnetic crystal phases are contained. The average size of 10nm or more 200nm or less and a soft magnetism phase has the average size of a hard magnetism phase within the limits of 1nm or more 100nm or less. For said hard magnetism phase, the $\text{R}_2\text{Fe}_{14}\text{B}$ mold compound phase which it is larger than said soft magnetism phase, and is a hard magnetism phase is the iron machine rare earth alloy magnet whose coercive force 60% or more of whole exists at the rate of a volume ratio, and is 480 or more kA/m.

[Claim 2] The iron machine rare earth alloy magnet according to claim 1 with which the presentation ratio x, and y and z are satisfied of $0.5 \leq z \leq 6 \text{at\%}$ $8 \leq y \leq 9.3 \text{at\%}$ $10 < x < 17 \text{at\%}$, respectively.

[Claim 3] The iron machine rare earth alloy magnet according to claim 1 with which the $\text{R}_2\text{Fe}_{14}\text{B}$ mold compound phase, the boride phase, and the $\alpha\text{-Fe}$ phase are intermingled in the same metal texture.

[Claim 4] The diameter of average crystal grain of said $\alpha\text{-Fe}$ phase and a boride phase is a 1nm or more iron machine rare earth alloy magnet according to claim 3 which is 50nm or less.

[Claim 5] Said boride phase is an iron machine rare earth alloy magnet containing ferromagnetic iron machine boride according to claim 4.

[Claim 6] Said iron machine boride is an iron machine rare earth alloy magnet according to claim 5 characterized by including Fe_3B and/or Fe_{23}B_6 .

[Claim 7] The iron machine rare earth alloy magnet according to claim 1 with which the presentation ratio x and z are satisfied of $z/x \geq 0.1$.

[Claim 8] Said soft magnetism phase is an iron machine rare earth alloy magnet according to claim 1 located in the grain boundary of said hard magnetism phase.

[Claim 9] The iron machine rare earth alloy magnet according to claim 1 whose presentation ratio y of said R is below 9.0 atom %.

[Claim 10] An iron machine rare earth alloy magnet given in either of claims 1-6 in which thickness has 10-micrometer or more thin band configuration of 300 micrometers or less.

[Claim 11] An iron machine rare earth alloy magnet given in either of claims 1-6 by which disintegration is carried out.

[Claim 12] The iron machine rare earth alloy magnet according to claim 11 whose mean particle diameter of a powder particle is 30 micrometers or more 250 micrometers or less.

[Claim 13] Coercive force $H_cJ \geq 480 \text{ kA/m}$, an iron machine rare earth alloy magnet given in either of claims 1-6 which have the hard magnetic property of residual magnetic flux density $B_r \geq 0.7 \text{ T}$.

[Claim 14] An iron machine rare earth alloy magnet given in either of claims 1-6 which have the hard magnetic property of residual magnetic flux density $B_r \geq 0.85T$, maximum energy product $(BH)_{max} \geq 120 \text{ kJ/m}^3$, and proper coercive force $H_cJ \geq 480 \text{ kA/m}$.

[Claim 15] The bond magnet which fabricated the magnet powder containing the powder of the iron machine rare earth alloy magnet indicated by claim 11 by resin.

[Claim 16] an empirical formula -- $100(\text{Fe}_{1-m}\text{Tm})_x\text{-y-zQxRyMz}$ (one or more sorts of elements chosen from the group which T becomes from Co and nickel --) One or more sorts of elements chosen from the group which Q becomes from B and C, one or more sorts of rare earth metal elements with which R does not contain La and Ce substantially, M is the metallic element chosen from the group which consists of Ti, Zr, and Hf. It is expressed by at least one sort of metallic elements which surely contain Ti. The presentation ratio x, y, z, and m $x < 10 < 17$ atom %, $6 \leq y \leq 9.3$ atom %, $0.1 \leq z \leq 6$ atom % and the process at which the molten metal of an alloy which satisfies $0 \leq m \leq 0.5$ is produced, and by quenching the molten metal of said alloy, respectively The cooling process which produces the quenching alloy which contains 60% or more of $\text{R}_2\text{Fe}_{14}\text{B}$ mold crystal phase at the rate of a volume ratio, Include the heat treatment process which heats said quenching alloy, and two or more kinds of ferromagnetic crystal phases are contained. The manufacture approach of an iron machine rare earth alloy magnet of the average size of 10nm or more 200nm or less and a soft magnetism phase having the average size of a hard magnetism phase within the limits of 1nm or more 100nm or less, and manufacturing an iron machine rare earth alloy magnet with said larger hard magnetism phase than said soft magnetism phase.

[Claim 17] Said cooling process is the manufacture approach of the iron machine rare earth alloy magnet according to claim 16 which quenches the molten metal of said alloy in the controlled atmosphere of 30 or more kPas of pressures, and includes producing the quenching alloy containing an $\text{R}_2\text{Fe}_{14}\text{B}$ mold compound

phase with a mean particle diameter of 50nm or less.

[Claim 18] Said cooling process is the manufacture approach of the iron machine rare-earth-alloy magnet according to claim 17 which includes the process which takes heat from the alloy in said supercooling condition, and grows said R₂Fe₁₄B mold compound phase with said controlled atmosphere after the process which forms the alloy which is contacted on the front face of the cooling roller turning around said alloy molten metal, and is in a supercooled liquid condition, and the alloy in said supercooling condition separate from a cooling roller.

[Claim 19] The manufacture approach of an iron machine rare earth alloy magnet according to claim 16 that the organization which contains three or more kinds of crystal phases which contain an R₂Fe₁₄B mold compound phase, an alpha-Fe phase, and a boride phase at least according to said heat treatment process is formed, and the diameter of average crystal grain of 150nm or less and said alpha-Fe phase of 20nm or more, and a boride phase is set to 1nm or more 50nm or less by the diameter of average crystal grain of said R₂Fe₁₄B mold compound phase.

[Claim 20] Said boride phase is the manufacture approach of the iron machine rare earth alloy magnet containing ferromagnetic iron machine boride according to claim 19.

[Claim 21] Said iron machine boride is the manufacture approach of the iron machine rare earth alloy magnet containing Fe₃B and/or Fe₂₃B₆ according to claim 20.

[Claim 22] The manufacture approach of the bond magnet which includes the process which prepares the powder of the iron machine rare earth alloy magnet produced by the manufacture approach of the iron machine rare earth alloy magnet a publication by either of claims 16-21, and the process which produces a bond magnet using the powder of said iron machine rare earth alloy magnet.

[Claim 23] The iron machine rare earth alloy magnet indicated by claims 11 or 12 by which surface treatment was performed to the powder front face.

[Claim 24] The bond magnet according to claim 15 with which surface preparation was performed.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the manufacture approach of an iron machine rare earth alloy magnet of having two or more ferromagnetic phases especially, about the manufacture approach of the permanent magnet used suitable for various motors or an actuator.

[0002]

[Description of the Prior Art] In recent years, in the device for household electric appliances, OA equipment, electronic autoparts, etc., much more high-performance-izing and small lightweight-ization are demanded. Therefore, about the permanent magnet used for these devices, it is required that making the engine-performance tare quantitative ratio as the whole magnetic circuit into max should be called for, for example, a residual magnetic flux density B_r should use the permanent magnet more than 0.5T (tesla). However, depending on the

conventional comparatively cheap hard ferrite magnet, a residual magnetic flux density B_r cannot be carried out more than 0.5T.

[0003] As a permanent magnet which has current and the high residual magnetic flux density B_r beyond 0.5T, the Sm-Co system magnet produced by powder-metallurgy processing is known. Except a Sm-Co system magnet, the Nd-Fe-B system magnet produced by powder-metallurgy processing and the Nd-Fe-B system quenching magnet produced by the melt quenching method can demonstrate the high residual magnetic flux density B_r . The former Nd-Fe-B system magnet is indicated by JP,59-46008,A, and the latter Nd-Fe-B system quenching magnet is indicated by JP,60-9852,A.

[0004]

[Problem(s) to be Solved by the Invention] however, any of Sm and Co from which a Sm-Co system magnet serves as a raw material -- although -- since it is expensive, it has the fault that a magnet price is high.

[0005] In the case of a Nd-Fe-B system magnet, since it is included, using cheap Fe as a principal component (60 % of the weight - about 70% of the weight of the whole), compared with a Sm-Co system magnet, it is cheap, but there is a problem that the costs which the production process takes are high. One of the reasons nil why production process costs are high is that a large-scale facility and a great process are needed for the separation purification and the reduction reaction of Nd for which a content occupies ten whole atoms % - 15 atom % extent. Moreover, when based on powder-metallurgy processing, the number of production processes surely increases.

[0006] On the other hand, since the Nd-Fe-B system quenching magnet manufactured by the melt quenching method is obtained at the comparatively easy process of a dissolution process -> liquid cooling process -> heat treatment process, it has the advantage that process costs are cheap compared with the Nd-Fe-B system magnet by powder-metallurgy processing. However, since it is necessary to mix with resin the magnet powder produced from the quenching alloy, and to form a bond magnet in order to obtain a bulk-like permanent magnet

when based on a melt quenching method, the filling factor (rate of a volume ratio) of the magnet powder occupied to the fabricated bond magnet is at most about 80%. Moreover, the quenching alloy produced with the melt quenching method is isotropy magnetically.

[0007] From the above reason, it has the problem that the Nd-Fe-B system quenching magnet manufactured using the melt quenching method has low B_r compared with the Nd-Fe-B system sintered magnet of the anisotropy manufactured with powder-metallurgy processing.

[0008] As the technique of improving the property of a Nd-Fe-B system quenching magnet, it is effective to add complexly at least a kind of element chosen from the group which consists of Zr, Nb, Mo, Hf, Ta, and W, and a kind of element chosen from the group which consists of Ti, V, and Cr at least as indicated by JP,1-7502,A. Although coercive force H_{cJ} and corrosion resistance improve by addition of such an element, the effective method of improving a residual magnetic flux density B_r is not learned besides improving the consistency of a bond magnet.

[0009] In the case of the Nd-Fe-B system magnet, the concentration of rare earth elements has a presentation low in comparison, i.e., the near presentation of $Nd_{3.8}Fe_{77.2}B_{19}$ (atomic %), and the magnet ingredient which makes a Fe_3B mold compound the main phase is proposed (J.de Phys, such as R.Coehoorn, C 8 and 1998, 669-670 pages). By performing heat-of-crystallization processing to the amorphous alloy produced with the melt quenching method, this permanent magnet ingredient has the metastable structure formed from the fine crystal aggregate with which the $Nd_2Fe_{14}B$ phase which is Fe_3B phase and hard magnetism which are soft magnetism is intermingled, and is called the "nano composite magnet." Although having the high residual magnetic flux density B_r beyond 1T is reported about such a nano composite magnet, the coercive force H_{cJ} is comparatively as low as 160 kA/m - 240 kA/m. Therefore, use of this permanent magnet ingredient is restricted to the application from which the magnetic operating point becomes one or more.

[0010] Moreover, various metallic elements are added into the raw material alloy of a nano composite magnet: although the attempt which raises magnetic properties is made (JP,3-261104,A --) A U.S. Pat. No. 4,836,868 number, JP,7-122412,A, The international public presentation official report WO 003/03403 of international application, wcChan, and et.al."THE EFFECTS OF REFRACTORY METALS ON THE MAGNETIC PROPERTIES OF α -Fe/R₂Fe₁₄B-TYPE NANOCOMPOSITES", IEEE, Trans.Magn.No.5, INTERMAG.99, Kyongju, Korea pp.3265-3267, 1999, Sufficient "characteristic value per cost" is not necessarily acquired.

[0011] This invention is made in view of the above-mentioned situation, and the place made into the purpose is to offer the manufacture approach of a permanent magnet that an iron machine alloy magnet with the outstanding magnetic properties with which are satisfied of residual magnetic flux density $B_r \geq 0.85T$ can be manufactured cheaply, maintaining high coercive force (for example, $H_cJ \geq 480$ kA/m).

[0012]

[Means for Solving the Problem] the iron machine rare earth alloy magnet by this invention -- an empirical formula -- $100(\text{Fe}_{1-m}\text{Tm})_x\text{-y-zQxRyMz}$ (one or more sorts of elements chosen from the group which T becomes from Co and nickel --) One or more sorts of elements chosen from the group which Q becomes from B and C, one or more sorts of rare earth metal elements with which R does not contain La and Ce substantially, M is the metallic element chosen from the group which consists of Ti, Zr, and Hf. It is expressed by at least one sort of metallic elements which surely contain Ti. The presentation ratio x, y, z, and m, respectively $10 < x \leq 20$ atom %, $y < 6 \leq 10$ atom %, $0.1 \leq z \leq 12$ atom % and $0 \leq m \leq 0.5$ are satisfied, two or more kinds of ferromagnetic crystal phases are contained, and the average size of 10nm or more 200nm or less and a soft magnetism phase has the average size of a hard magnetism phase within the limits of 1nm or more 100nm or less.

[0013] In a certain desirable operation gestalt, the presentation ratio x, and y and

z satisfy $0.5 \leq z \leq 6 \text{at\%}$ $8 \leq y \leq 9.3 \text{at\%}$ $10 < x < 17 \text{at\%}$, respectively.

[0014] In a certain desirable operation gestalt, the $R_2Fe_{14}B$ mold compound phase, the boride phase, and the $\alpha\text{-Fe}$ phase are intermingled in the same metal texture.

[0015] In a certain desirable operation gestalt, the diameter of average crystal grain of said $\alpha\text{-Fe}$ phase and a boride phase is 1nm or more 50nm or less.

[0016] In a certain desirable operation gestalt, said boride phase contains ferromagnetic iron machine boride.

[0017] In a certain desirable operation gestalt, said iron machine boride contains Fe_3B and/or $Fe_{23}B_6$.

[0018] In a certain desirable operation gestalt, the presentation ratio x and z satisfy $z/x \geq 0.1$.

[0019] In a certain desirable operation gestalt, the presentation ratio y of said R is below 9.5 atom %.

[0020] In a certain desirable operation gestalt, the presentation ratio y of said R is below 9.0 atom %.

[0021] In a certain desirable operation gestalt, thickness has 10-micrometer or more thin band configuration of 300 micrometers or less.

[0022] Disintegration of said iron machine rare earth alloy magnet may be carried out. In this case, as for the mean particle diameter of a powder particle, it is desirable that it is [30 micrometer or more] 250 micrometers or less.

[0023] In a certain desirable operation gestalt, coercive force $H_cJ \geq 480 \text{ kA/m}$ and the magnet which has the hard magnetic property of residual magnetic flux density $Br \geq 0.7T$ are obtained.

[0024] Furthermore, with a desirable operation gestalt, the magnet which has the hard magnetic property of residual magnetic flux density $Br \geq 0.85T$, maximum energy product $(BH)_{\text{max}} \geq 120 \text{ kJ/m}^3$, and proper coercive force $H_cJ \geq 480 \text{ kA/m}$ is obtained.

[0025] The bond magnet by this invention fabricates the magnet powder containing the powder of the above-mentioned iron machine rare earth alloy

magnet by resin.

[0026] The quenching alloy for iron machine rare earth alloy magnets by this invention an empirical formula -- $100(\text{Fe}_{1-m}\text{Tm})_x\text{-y-zQxRyMz}$ (one or more sorts of elements chosen from the group which T becomes from Co and nickel --) One or more sorts of elements chosen from the group which Q becomes from B and C, one or more sorts of rare earth metal elements with which R does not contain La and Ce substantially, M is the metallic element chosen from the group which consists of Ti, Zr, and Hf. It is expressed by at least one sort of metallic elements which surely contain Ti, and the presentation ratio x, and y, z and m satisfy $10 < x \leq 20$ atom %, $y \leq 6$ atom %, $0.1 \leq z \leq 12$ atom %, and $0 \leq m \leq 0.5$, respectively.

[0027] In a certain desirable operation gestalt, excluding α -Fe substantially, it had the organization containing an $\text{R}_2\text{Fe}_{14}\text{B}$ mold compound phase and an amorphous phase, and said $\text{R}_2\text{Fe}_{14}\text{B}$ mold compound phase forms 60% or more of the whole at the rate of a volume ratio.

[0028] With a certain desirable operation gestalt, $10 < x < 17\text{at\%}$, $8 \leq y \leq 9.3\text{at\%}$ and $0.5 \leq z \leq 6\text{at\%}$ is satisfied, and an $\text{R}_2\text{Fe}_{14}\text{B}$ mold compound phase with a mean particle diameter of 50nm or less is contained more than 60 volume %.

[0029] The quenching alloy for iron machine rare-earth-alloy magnets by this invention is produced by cooling the alloy molten metal containing Fe, Q (one or more sorts of elements chosen from the group which Q becomes from B and C), R (R being rare earth elements), and Ti, and has the organization which starts the growth of a compound crystal phase which has the $\text{R}_2\text{Fe}_{14}\text{B}$ mold crystal structure before growth initiation of an α -Fe crystal phase by heat treatment including the amorphous phase.

[0030] The manufacture approach of the iron machine rare earth alloy magnet by this invention The process which produces the alloy molten metal containing Fe, Q (one or more sorts of elements chosen from the group which Q becomes from B and C), R (R is rare earth elements), and Ti, The process which makes the growth with the $\text{R}_2\text{Fe}_{14}\text{B}$ mold crystal structure of a compound crystal phase

start, and makes growth of an alpha-Fe crystal phase start after that is included by cooling said alloy molten metal and heating the process which produces the coagulation alloy containing an amorphous phase, and said coagulation alloy.

[0031] With a certain desirable operation gestalt, said alloy molten metal is cooled using the strip cast method.

[0032] The manufacture approach of the iron machine rare earth alloy magnet by this invention an empirical formula -- $100(\text{Fe}_{1-m}\text{Tm})_{-x-y-z}\text{QxRyMz}$ (one or more sorts of elements chosen from the group which T becomes from Co and nickel --) One or more sorts of elements chosen from the group which Q becomes from B and C, one or more sorts of rare earth metal elements with which R does not contain La and Ce substantially, M is the metallic element chosen from the group which consists of Ti, Zr, and Hf. It is expressed by at least one sort of metallic elements which surely contain Ti. The presentation ratio x, y, z, and m $10 < x \leq 20$ atom %, $y < 6 \leq 10$ atom %, $0.1 \leq z \leq 12$ atom % and the process at which the molten metal of an alloy which satisfies $0 \leq m \leq 0.5$ is produced, and by quenching the molten metal of said alloy, respectively The cooling process which produces the quenching alloy with which an $\text{R}_2\text{Fe}_{14}\text{B}$ mold crystal phase and an amorphous phase are intermingled, Said quenching alloy is crystallized, two or more kinds of ferromagnetic crystal phases are contained by it, and the average size of a hard magnetism phase includes the process in which the average size of 10nm or more 200nm or less and a soft magnetism phase forms the organization which is within the limits of 1nm or more 100nm or less.

[0033] With a certain desirable operation gestalt, the quenching alloy which contains 60% or more of $\text{R}_2\text{Fe}_{14}\text{B}$ mold compound phase at the rate of a volume ratio is produced in said cooling process.

[0034] In a certain desirable operation gestalt, said cooling process quenches the molten metal of said alloy in the controlled atmosphere of 30 or more kPas of pressures, and includes producing the quenching alloy containing an $\text{R}_2\text{Fe}_{14}\text{B}$ mold compound phase with a mean particle diameter of 50nm or less.

[0035] The front face of the cooling roller with which said cooling process rotates

said alloy molten metal in a certain desirable operation gestalt is made to contact, after the process which forms the alloy in a supercooled liquid condition, and the alloy in said supercooling condition separate from a cooling roller, heat is taken from the alloy in said supercooling condition with said controlled atmosphere, and the process which grows said R₂Fe₁₄B mold compound phase is included.

[0036] With a certain desirable operation gestalt, heat-of-crystallization processing is performed to said quenching alloy. The organization containing three or more kinds of crystal phases which contain an R₂Fe₁₄B mold compound phase, an α -Fe phase, and a boride phase at least is formed. The process which sets the diameter of average crystal grain of 150nm or less and said α -Fe phase of 20nm or more, and a boride phase to 1nm or more 50nm or less for the diameter of average crystal grain of said R₂Fe₁₄B mold compound phase is included.

[0037] In a certain desirable operation gestalt, said boride phase contains ferromagnetic iron machine boride.

[0038] In a certain desirable operation gestalt, said iron machine boride contains Fe₃B and/or Fe₂₃B₆.

[0039] With a certain desirable operation gestalt, the molten metal of said alloy is cooled using the strip cast method.

[0040] The manufacture approach of the bond magnet by this invention includes the process which prepares the powder of the iron machine rare earth alloy magnet produced by the manufacture approach of the iron machine rare earth alloy magnet one of the above, and the process which produces a bond magnet using the powder of said iron machine rare earth alloy magnet.

[0041] With a certain desirable operation gestalt, surface treatment is performed on the surface of powder.

[0042] With a certain desirable operation gestalt, surface treatment is performed to the bond magnet.

[0043]

[Embodiment of the Invention] The iron machine rare earth alloy magnet of this

invention cools the molten metal of the rare earth-iron-boron system alloy containing Ti, and is formed from the quenching alloy solidified by it. Although this rapid solidification alloy contains a crystal phase, it is heated if needed and crystallization is advanced further.

[0044] By adding Ti to the iron machine rare earth alloy which has the presentation of the specific range, this invention person controls a deposit and growth of the alpha-Fe phase which is easy to produce in the cooling process of an alloy molten metal, and came to hit preferential and that homogeneity can be advanced on an idea of a header and this invention for the crystal growth of the $R_2Fe_{14}B$ mold compound phase which bears hard magnetic property.

[0045] When Ti is not added, an alpha-Fe phase deposits in advance of a deposit and growth of $Nd_2Fe_{14}B$ phase, and it is easy to grow up. Therefore, in the phase which the heat-of-crystallization processing to a quenching alloy completed, the alpha-Fe phase of soft magnetism will make it big and rough.

[0046] On the other hand, when Ti is added, in order that the kinetics (kinetics) of a deposit and growth of an alpha-Fe phase may become late and a deposit and growth may take time amount to it, before a deposit and growth of an alpha-Fe phase are completed, it is thought that a deposit and growth of $Nd_2Fe_{14}B$ phase begin. For this reason, before an alpha-Fe phase makes it big and rough, $Nd_2Fe_{14}B$ phase grows up to be the condition of having distributed to homogeneity greatly. It seems that moreover, the compatibility of Ti over B is strong and it is easy to be condensed in iron machine boride. When Ti and B join together strongly within iron machine boride, it is thought that Ti addition stabilizes iron machine boride.

[0047] according to this invention, soft magnetism phases, such as iron machine boride and an alpha-Fe phase, are made detailed by work of Ti -- both, $Nd_2Fe_{14}B$ phase distributes to homogeneity, and, moreover, the rate of a volume ratio of $Nd_2Fe_{14}B$ phase increases. Consequently, coercive force and magnetization (residual magnetic flux density) increase, and the square shape nature of a demagnetization curve improves.

[0048] Hereafter, the iron machine rare earth alloy magnet of this invention is explained more to a detail.

[0049] As for the iron machine rare earth alloy magnet of this invention, the empirical formula is suitably expressed by $100(\text{Fe}_{1-m}\text{Tm})_x\text{-y-zQxRyMz}$. One or more sorts of elements chosen from the group which T becomes from Co and nickel here, one or more sorts of elements chosen from the group which Q becomes from B (boron) and C (carbon), one or more sorts of rare earth metal elements with which R does not contain La and Ce substantially, and M are at least one sort of metallic elements chosen from the group which consists of Ti, Zr, and Hf, and surely contain Ti.

[0050] As for x which specifies a presentation ratio, and y, z and m, it is desirable respectively to satisfy $10 < x \leq 20$ atom %, $y < 6 \leq 10$ atom %, $0.1 \leq z \leq 12$ atom %, and the relation of $0 \leq m \leq 0.5$.

[0051] Level with the iron machine rare earth alloy magnet of this invention equivalent to the case where magnetization (residual magnetic flux density) does not add Ti by addition of Ti although the presentation ratio of rare earth elements is under the whole 10 atom % is maintained, or it increases, and the unexpected effectiveness that the square shape nature of a demagnetization curve improves is demonstrated.

[0052] With the iron machine rare earth alloy magnet of this invention, since the size of a soft magnetism phase is detailed, even if each configuration phase joins together by the exchange interaction and iron machine boride and a soft magnetism phase like $\alpha\text{-Fe}$ exist in addition to the $\text{R}_2\text{Fe}_{14}\text{B}$ mold compound phase of hard magnetism, it becomes possible to show the square shape nature of the demagnetization curve which was excellent as the whole alloy.

[0053] The iron machine rare earth alloy magnet of this invention contains suitably the iron machine boride and $\alpha\text{-Fe}$ which have the saturation magnetization of an $\text{R}_2\text{Fe}_{14}\text{B}$ mold compound phase, an EQC, or saturation magnetization higher than it. This iron machine boride is for example, Fe_3B (saturation magnetization 1.5T) and Fe_{23}B_6 (saturation magnetization 1.6T).

Here, the saturation magnetization of $R_2Fe_{14}B$ is about 1.6T, and the saturation magnetization of α -Fe is 2.1T.

[0054] Usually, although $R_2Fe_{23}B_3$ is generated when the presentation ratio x of B exceeds 10 atom % and the presentation ratio y of rare earth elements R is moreover in the range below 8 atom % more than 6 atom % Even if it is the case where the raw material alloy in such presentation range is used, iron machine borides, such as $R_2Fe_{14}B$, and $Fe_{23}B_6$, Fe_3B , are generable instead of $R_2Fe_{23}B_3$ by adding Ti like this invention. Such iron machine borides are contributed to the improvement in magnetization.

[0055] According to the experiment of this invention person, only when Ti was added, it turned out that unlike the case where the metal of other classes, such as V, Cr, Mn, Nb, and Mo, is added the fall of magnetization does not arise but magnetization improves rather for the first time. Moreover, when Ti was added, compared with other above-mentioned alloying elements, the square shape nature of a demagnetization curve became good especially.

[0056] Moreover, such Ti addition effectiveness is notably demonstrated, when B concentration exceeds 10at(s)%. Hereafter, this point is explained, referring to drawing 1 .

[0057] Drawing 1 is a graph which shows the relation of the maximum MAG energy-product (BH) max and boron concentration of the Nd-Fe-B magnet with which Ti is not added. A white bar shows the data of the sample containing 10 - 14at% Nd among a graph, and the black bar shows the data of the sample containing 8 - 10at% Nd. On the other hand, drawing 2 is a graph which shows the relation of the maximum MAG energy-product (BH) max and boron concentration of the Nd-Fe-B magnet with which Ti was added. A white bar shows the data of the sample containing 10 - 14at% Nd among a graph, and the black bar shows the data of the sample containing 8 - 10at% Nd.

[0058] By the sample by which Ti is not added, maximum MAG energy-product (BH) max is falling as boron increases irrespective of the content of Nd exceeding 10at(s)%, so that drawing 1 may show. Extent of this fall is large by

the case where the content of Nd is 10 - 14at%. Such an inclination is known from the former and it has been thought desirable in the permanent magnet which makes Nd₂Fe₁₄B phase the main phase to set the amount of boron as less than [10at%]. For example, in a U.S. Pat. No. 4,836,868 number, boron concentration indicates a 5 - 9.5at% example, and less than [more than 4at%12at%] is taught as range where boron concentration is still more desirable, and it is teaching the range not more than more than 4at%10at% as more desirable range.

[0059] On the other hand, by the sample by which Ti was added, maximum MAG energy-product (BH) max is improving in a certain range in which boron exceeds 10at(s)% so that drawing 2 may show. This improvement is remarkable especially when the content of Nd is 8 - 10at%.

[0060] Thus, according to this invention, if B concentration exceeds 10at(s)%, it will become possible to acquire the effectiveness which cannot be expected by Ti addition from the Prior-art common sense that magnetic properties deteriorate.

[0061] Next, the manufacture approach of the iron machine rare earth alloy magnet by this invention is explained.

[0062] According to the manufacture approach of this invention, the molten metal of an iron machine alloy which has the aforementioned presentation is cooled in an inert atmosphere, and the quenching alloy which contains an R₂Fe₁₄B mold compound phase more than the whole 60 volume % by it is produced. The average size of the R₂Fe₁₄B mold compound phase in a quenching alloy is 80nm or less. If it heat-treats to this quenching alloy if needed, the amorphous substance which remained in the quenching alloy can be crystallized.

[0063] With a desirable operation gestalt, the above-mentioned alloy molten metal is cooled in the ambient atmosphere of 30 or more kPas of pressures. Thereby, even after separating from a cooling roller, in response to the secondary-cooling-of-concrete effectiveness by the controlled atmosphere, it contact to a cooling roller not only quenches an alloy molten metal, but is cooled appropriately.

[0064] It is possible to control the condition of the alloy at the time of separating from a cooling roller by adjusting the rotation peripheral velocity of a cooling roller appropriately in the supercooled liquid condition, after the alloy of a supercooling condition separates from a cooling roller, by the controlled atmosphere, cooling of it is carried out and it is crystallized.

[0065] The secondary-cooling-of-concrete effectiveness by the atmospheric-air controlled atmosphere is explained referring to drawing 3. Drawing 3 is a graph which shows typically the relation between the elapsed time (Time) from cooling process initiation, and alloy temperature (Temperature). The cooling path b of an alloy in case [relatively low] the cooling path a and ambient-gas-pressure force of an alloy in case [relatively high] the ambient-gas-pressure force exceeds 30kPa(s) are less than 30kPa(s) is shown in the graph, it combines, and the deposit field of an alpha-Fe phase, Nd₂Fe₁₄ B phase, and a Fe₂₃ B₆ phase is illustrated. T_m is the melting point of an alloy here and T_g is the glass transition temperature of an alloy.

[0066] Since the big secondary-cooling-of-concrete effectiveness by the controlled atmosphere cannot be expected when the pressure of a controlled atmosphere is low (cooling path b) that drawing 3 shows, rotational speed of a cooling roller is made high and the rate of quenching (primary cooling of concrete) by the cooling roller is relatively made high. After an alloy separates from the front face of a cooling roller, it is relatively cooled at a late rate by the controlled atmosphere (secondary cooling of concrete). The point with which the cooling path b has bent corresponds, when an alloy exfoliates from a cooling roller.

[0067] On the other hand, when the pressure of a controlled atmosphere is relatively high (cooling path a), the cooling (secondary cooling of concrete) effectiveness by the controlled atmosphere becomes strong, and the time amount which passes through the field where Nd₂Fe₁₄ B phase is generated is shortened. For this reason, growth of Nd₂Fe₁₄ B phase is controlled and that from which detailed Nd₂Fe₁₄ B phase is obtained is presumed.

[0068] Thus, if the ambient-gas-pressure force is made low so that it may be less than 30kPa, in order that the diameter of crystal grain of the R₂Fe₁₄B mold compound phase generated in a quenching alloy may make it big and rough, the magnet property finally acquired will deteriorate.

[0069] On the contrary, if the pressure of a controlled atmosphere becomes high too much exceeding ordinary pressure (atmospheric pressure), the effect of the controlled atmosphere wound between an alloy molten metal and a cooling roller will become large, and sufficient cooling by the cooling roller will no longer be attained. Consequently, big and rough alpha-Fe deposits and good hard magnetic property is no longer obtained.

[0070] According to the experiment of this invention person, as for the pressure of a controlled atmosphere, it is desirable at the time of quenching to control by 30 or more kPas moreover below at ordinary pressure (101.3kPa), and it is still more desirable to it to make it the 30 or more kPas range of 90 or less kPa. The more desirable range is 40 or more-kPa 60 or less kPa.

[0071] Under the above-mentioned ambient-gas-pressure force, the range where roll surface peripheral velocity is desirable is 50m/second or less more than 4m (m/second)/second. When roll surface peripheral velocity becomes slower 4m /than a second, the crystal grain of the R₂Fe₁₄B mold compound phase contained in a quenching alloy will make it big and rough. Consequently, by heat treatment, an R₂Fe₁₄B mold compound phase may become still larger, and magnetic properties may deteriorate.

[0072] On the other hand, if roll surface peripheral velocity becomes quicker 50m /than a second, it becomes amorphous and an R₂Fe₁₄B mold compound phase will be in the condition with a nearly perfect quenching alloy of hardly depositing. Therefore, in heat-of-crystallization processing, grain growth of an R₂Fe₁₄B mold compound phase is remarkable, and in order that an organization may ununiformity-ize, improvement in magnetic properties is not attained.

[0073] According to the experiment, the still more desirable range of roll surface peripheral velocity is 5m/second or more 30m/second or less, and the still more

desirable range is 5m/second or more 20m/second or less.

[0074] In addition, in this invention, most big and rough alpha-Fe is not deposited in a quenching alloy, and the organization which has a detailed R2Fe14B mold compound phase or the organization which has a detailed R2Fe14B mold compound phase, and the organization where the amorphous phase was intermingled are produced. Thereby, the compound-die permanent magnet of the condition which soft magnetism phases, such as an iron machine boride phase, distributed minutely between hard magnetism phases (grain boundary), or the high performance which exists by the extended state thinly can be obtained after heat treatment. In addition, not only the phase constituted by only the part in which atomic arrangement carried out disordering to the "amorphous phase" in this specification completely but the phase which contains partially the precursor, the microcrystal (size: several nm or less), or the atomic cluster of crystallization shall be included. Specifically, the phase which can identify the crystal structure clearly neither by the X diffraction nor transmission electron microscope observation will be widely called an "amorphous phase."

[0075] If it was going to produce conventionally a quenching alloy which cools the alloy molten metal which has the presentation which is similar so that the target presentation of this invention, and contains an R2Fe14B mold compound phase more than 60 volume %, since the alloy organization where alpha-Fe deposited mostly would be obtained, there was a problem that alpha-Fe will make it big and rough by subsequent heat-of-crystallization processing. If soft magnetism phases, such as alpha-Fe, make it big and rough, a magnet property will deteriorate greatly and the permanent magnet which is equal to practical use will not be obtained at all.

[0076] Like the raw material alloy presentation used especially by this invention, since it was the high amorphous generation ability which boron has when there are comparatively many contents of boron, even if it made the cooling rate of an alloy molten metal late, the crystal phase was hard to be generated. Therefore, if it is going to produce a rapid solidification alloy with which the cooling rate of an

alloy molten metal is fully reduced, and the rate of a volume ratio of an R₂Fe₁₄B mold compound phase exceeds 60% according to the conventional technique, many alpha-Fe or its precursor deposited in addition to the R₂Fe₁₄B mold compound phase, by subsequent heat-of-crystallization processing, big and rough-ization of an alpha-Fe phase would advance, and the magnet property will have deteriorated greatly.

[0077] In order to have increased the coercive force of a nano composite magnet conventionally from the above thing, the cooling rate of an alloy molten metal was raised, and after changing into the condition that most rapid solidification alloys are occupied with an amorphous phase, common sense that it is desirable forming the organization made detailed by homogeneity by heat-of-crystallization processing from the amorphous phase existed. this should perform crystallization from an amorphous phase by the heat treatment process which is easy to control, in order to obtain a nano composite with the alloy organization which the detailed crystal phase distributed -- ** -- it is because it thought.

[0078] For this reason, after producing the rapid solidification alloy which makes an amorphous phase the main phase by adding La excellent in amorphous generation ability into a raw material alloy, and quenching the molten metal of that raw material alloy, Both Nd₂Fe₁₄ B phase and an alpha-Fe phase are deposited and grown up by heat-of-crystallization processing. The technique which makes [about dozens of nm] any phase detailed is reported ().

[wcChan,] [et.al."THE EFFECTS OF REFRACTORY METALS ON THE MAGNETIC PROPERTIES] OF alpha-Fe/R₂Fe₁₄B-TYPE NANOCOMPOSITES", IEEE, Trans.Magn.No.5, INTERMAG.99, Kyongju, Korea pp.3265-3267, 1999. In addition, this paper is teaching the desirable thing, when that minute amount addition (2at%) of refractory metal elements, such as Ti, raises a magnet property and making the presentation ratio of Nd which is rare earth elements increase from 9.5at(s)% to 11.0at(s)% make detailed both Nd₂Fe₁₄ B phase and an alpha-Fe phase. Addition of the above-mentioned refractory metal controls generation of boride (R₂Fe₂₃B₃ and Fe₃B), and it is

performed in order to produce the magnet which consists only of 2 of Nd₂Fe₁₄B phase and an alpha-Fe phase phases.

[0079] On the other hand, in this invention, by work of Addition Ti, the deposit of an alpha-Fe phase can be suppressed at a rapid solidification process, and big and rough-ization of soft magnetism phases, such as iron machine boride in heat-of-crystallization down stream processing and an alpha-Fe phase, can be controlled further.

[0080] According to this invention, using a raw material alloy with comparatively few (less than [9.3at%]) amounts of rare earth elements, magnetization (residual magnetic flux density) and coercive force are high, and the permanent magnet excellent also in the square shape nature of a demagnetization curve can be manufactured.

[0081] As mentioned above, the increment in coercive force of the magnet by this invention is realized by having controlled big and rough-ization of a soft magnetism phase, depositing and growing up Nd₂Fe₁₄B phase preferentially at a cooling process, and making the rate of a volume ratio of Nd₂Fe₁₄B phase increase by it. moreover, the boron to which the increment in magnetization exists in a rapid solidification alloy by work of Ti -- boride phases, such as ferromagnetic iron machine boride, are generated from a rich nonmagnetic amorphous phase, and it is thought that it was obtained since the rate of a volume ratio of the nonmagnetic amorphous phase which remains after heat-of-crystallization processing was decreased.

[0082] It is desirable to form the organization containing three or more kinds of crystal phases which perform heat-of-crystallization processing and contain an R₂Fe₁₄B mold compound phase, a boride phase, and an alpha-Fe phase to the quenching alloy obtained as mentioned above if needed. During this organization, the diameter of average crystal grain of an R₂Fe₁₄B mold compound phase adjusts heat treatment temperature and time amount so that the diameter of average crystal grain of 200nm or less and the boride phase of 10nm or more, and an alpha-Fe phase may be set to 1nm or more 50nm or less. Although the

diameter of average crystal grain of an R₂Fe₁₄B mold compound phase is usually set to 30nm or more, it is set to 50nm or more depending on conditions. The diameter of average crystal grain of soft magnetism phases, such as a boride phase and an alpha-Fe phase, is set to 30nm or less in many cases, and becomes only the magnitude of several nm typically.

[0083] The diameter of average crystal grain of the R₂Fe₁₄B mold compound phase in a final magnet is larger than the diameter of average crystal grain of an alpha-Fe phase. Drawing 4 shows the metal texture of this magnet in the ** type. The detailed soft magnetism phase distributes and exists between big R₂Fe₁₄B mold compound phases relatively so that drawing 4 may show. Thus, since the average size of a soft magnetism phase is small enough, each configuration phase joins together by the exchange interaction, consequently the magnetization direction of a soft magnetism phase is restrained by the hard magnetism phase even if it is the case that the average size of an R₂Fe₁₄B mold compound phase is comparatively large, it becomes possible to show the square shape nature of the demagnetization curve which was excellent as the whole alloy.

[0084] It is thought that it is for combining with the element of the others [boron / this] in heat-of-crystallization processing since the amorphous phase which exists in a quenching alloy if the coagulation alloy whose R₂Fe₁₄B mold compound phase, as for the reason boride is easy to be generated as mentioned above when based on the manufacture approach of this invention, occupies most is produced will surely contain boron superfluously, and depositing and becoming easy to grow up. However, if the boron contained in the amorphous phase before heat treatment and other elements join together and the low compound of magnetization is generated, magnetization will fall as the whole magnet.

[0085] According to the experiment of this invention person, only when Ti was added, it turned out that unlike the case where the metal of other classes, such as V, Cr, Mn, Nb, and Mo, is added the fall of magnetization does not arise but magnetization improves rather. Moreover, when M (especially Ti) was added,

compared with other above-mentioned alloying elements, the square shape nature of a demagnetization curve became good especially. When controlling generation of the low boride of magnetization from these things, Ti is considered to carry out important work especially. Especially when there are little boron and Ti among the presentation range of the raw material alloy used by this invention in comparison, the iron machine boride phase which has ferromagnetism by heat treatment tends to deposit. In this case, since the rate of a volume ratio of a nonmagnetic amorphous phase which remains after heat-of-crystallization processing decreases as a result of incorporating the boron contained in a nonmagnetic amorphous phase in iron machine boride, and a ferromagnetic crystal phase increases, it is thought that a residual magnetic flux density B_r improves.

[0086] Hereafter, this point is explained more to a detail, referring to drawing 5 .

[0087] Drawing 5 is drawing showing typically the change of a detailed organization in the crystallization process of the rapid solidification alloy at the time of replacing with Ti and adding Nb etc., when Ti is added. When Ti is added, grain growth of each configuration phase is controlled in the temperature field higher than the temperature to which alpha-Fe deposits, and the outstanding hard magnetic property is maintained. On the other hand, when metallic elements, such as Nb, V, and Cr, are added, as a result of grain growth of each configuration phase advancing remarkably in a comparatively high temperature field in which alpha-Fe deposits and the switched connection of each configuration phase becoming weaker, the square shape nature of a demagnetization curve falls greatly.

[0088] First, the case where Nb, Mo, and W are added is explained. In this case, if it heat-treats in the comparatively low temperature field in which alpha-Fe does not deposit, it is possible to obtain the good hard magnetic property excellent in the square shape nature of a demagnetization curve. However, with the alloy which heat-treated at such temperature, it is presumed that the $R_2Fe_{14}B$ mold fine crystal phase distributes and exists in a nonmagnetic amorphous phase, and

the configuration of a nano composite magnet is not formed. Furthermore, if it heat-treats at high temperature, an alpha-Fe phase deposits out of an amorphous phase. Unlike the case where Ti is added, after a deposit, this alpha-Fe phase grows rapidly and is made big and rough. For this reason, the switched connection of each configuration interphase becomes weak, and the square shape nature of a demagnetization curve will deteriorate greatly.

[0089] On the other hand, when Ti is added, by heat treatment, the nano composite structure containing an R₂Fe₁₄B mold crystal phase, an iron machine boride phase, an alpha-Fe phase, and an amorphous phase is acquired, and it is made detailed to each configuration phase or homogeneity. Moreover, when Ti is added, growth of an alpha-Fe phase is controlled.

[0090] Since these addition metals dissolve to Fe and combine with Fe in antiferromagnetism when V and Cr are added, magnetization will fall greatly. Moreover, when V and Cr are added, the grain growth accompanying heat treatment is not fully controlled, but the square shape nature of a demagnetization curve deteriorates.

[0091] Thus, only when Ti is added, big and rough-ization of an alpha-Fe phase is controlled appropriately, and it becomes possible to form ferromagnetic iron machine boride. Furthermore, in order for Ti to delay crystallization of Fe primary phase (gamma-Fe which metamorphoses into alpha-Fe behind) at the time of liquid quenching, to consider as the element which makes generation of a supercooled liquid easy and to carry out important work with boron and carbon, It becomes possible to produce the quenching alloy with which an R₂Fe₁₄B mold crystal phase and an amorphous phase are intermingled, without depositing alpha-Fe greatly, even if it makes the cooling rate at the time of quenching an alloy molten metal into the comparatively low value of 102 degrees C/second - about 105 degrees C/second. Especially this is important for low-cost-izing in order to enable adoption of the strip cast method for having been suitable for mass production out of various melt quenching methods.

[0092] As an approach of quenching an alloy molten metal and obtaining a raw

material alloy, the strip cast method for pouring out a molten metal from tundish on a cooling roller directly, without performing control of flow of the molten metal by the nozzle orifice has high productivity, and is an approach that a manufacturing cost is low. In order to make amorphous the molten metal of a R-Fe-B system rare earth alloy in the cooling rate range which can be attained also by the strip cast method, it is usually necessary to add B (boron) more than 10 atom %. Thus, since the Nd₂Fe₁₄B three phase circuit which is the others, big and rough alpha-Fe, and the soft magnetism phase of un-nature magnetism deposits after performing heat-of-crystallization processing to a quenching alloy when many B is added, a homogeneous fine crystal organization is not obtained.

[phase / amorphous] Consequently, the rate of a volume ratio of a ferromagnetic phase falls, and the sharp fall of coercive force is invited by the fall of magnetization, and decline in the rate of an abundance ratio of Nd₂Fe₁₄B phase. However, if Ti is added like this invention, since the phenomenon mentioned above will be observed, magnetization improves unexpectedly.

[0093] In addition, a high thing tends to acquire a more final [a magnet / be /, rather than the case where a quenching alloy contains many amorphous phases / it / in the condition that many Nd₂Fe₁₄B phases are included] property. As for the rate of a volume ratio of the Nd₂Fe₁₄B phase occupied in a rapid solidification alloy, becoming more than 60 volume % is specifically desirable more than the whole one half. The value these 60 volume % is measured by Messbauer spectrum spectroscopy.

[0094] The whole quantity consists of B (boron), or the [reason for limitation of a presentation] Q consists of combination of B and C (carbon). As for the rate rate of an atomic ratio of C to the total amount of Q, it is desirable that it is 0.25 or less.

[0095] If the presentation ratio x of Q becomes below 10 atom %, it becomes difficult to produce the quenching alloy with which an R₂Fe₁₄B mold crystal phase and an amorphous phase are intermingled, and when the cooling rate at the time of quenching is comparatively low in 10² degrees C/second - about 10⁵

degrees C/[a second and], even if it heat-treats after that, only H_{cJ} of less than 480 kA/m will be obtained. Moreover, also in a melt quenching method, it becomes impossible for process costs to adopt the comparatively cheap strip cast method, and the price of a permanent magnet will rise. On the other hand, if the presentation ratio x of Q exceeds 20 atom %, since the rate of an abundance ratio of α -Fe which has the highest saturation magnetization in a configuration phase to the increase of the rate of the volume ratio of the amorphous phase in which after heat-of-crystallization processing remains, and coincidence will decrease, a residual magnetic flux density B_r will fall. It is desirable to set up so that the presentation ratio x of Q may exceed 10 atom % and it may consist of the above thing below 20 atom %. The range of the more desirable presentation ratio x is below 17 atom % more than 10 atom %.

[0096] R is one or more sorts of elements chosen from the group of rare earth elements (Y is included). If La or Ce exists, since coercive force and square shape nature will deteriorate, it is desirable that La and Ce are not included substantially. However, when La and Ce (below 0.5 atom %) of a minute amount exist as an impurity mixed unescapable, it is satisfactory on magnetic properties. Therefore, when it contains La and Ce below 0.5 atom %, it can be said that neither La nor Ce is included substantially.

[0097] As for R , it is desirable that Pr or Nd is included as an essential element, and, more specifically, it may permute a part of the essential element by Dy and/or Tb . When the presentation ratio y of R becomes under the whole 6 atom %, the compound phase which has the $R_2Fe_{14}B$ mold crystal structure required for the manifestation of coercive force does not fully deposit, but it becomes impossible to acquire the coercive force H_{cJ} of 480 or more kA/m. Moreover, if the presentation ratio y of R becomes more than 10 atom %, the iron machine boride which has ferromagnetism, and the abundance of α -Fe will fall. Therefore, as for the presentation ratio y of rare earth elements R , it is desirable to adjust the more than range under of 10 atom %, for example, 6 atom %, below to 9.5 atom % more than 6 atom %. The range of more desirable

R is below 9.3 atom % more than 8 atom %, and the range of most desirable R is below 9.0 atom % more than 8.3 atom %.

[0098] The addition metallic element M makes Ti indispensable, and may contain Zr and/or Hf further. Ti is an indispensable element in order to acquire the effectiveness mentioned above, it contributes to the improvement of the improvement in coercive force H_{cJ} and a residual magnetic flux density B_r , and the square shape nature of a demagnetization curve, and raises maximum energy product $(BH)_{max}$.

[0099] If the presentation ratio z of a metallic element M becomes under the whole 0.5 atom %, the effectiveness of Ti addition will not fully be discovered. On the other hand, if the presentation ratio z of a metallic element M exceeds the whole 12 atom %, since the rate of a volume ratio of the amorphous phase in which after heat-of-crystallization processing remains will increase, it is easy to invite the fall of a residual magnetic flux density B_r . As for the presentation ratio z of the above thing to the metallic element M, it is desirable to consider as the range below 12 atom % more than 0.5 atom %. The minimum of the range of more desirable z is 1.0 atoms %, and the upper limit of the range of more desirable z is 8.0 atoms %. Furthermore, the upper limit of the range of desirable z is 6.0 atoms %.

[0100] Moreover, since the amorphous phase which contains Q (for example, boron) superfluously is easy to be formed so that the presentation ratio x of Q is high, it is desirable to make high the presentation ratio z of a metallic element M. It is specifically desirable to adjust a presentation ratio so that $z/x \geq 0.1$ may be satisfied, and it is more desirable to satisfy $z/x \geq 0.15$.

[0101] In addition, in order that Ti may carry out desirable work especially, as for a metallic element M, it is desirable that Ti is surely included. In this case, it is desirable that it is [of Ti to the whole metallic element M] 70% or more comparatively (rate of an atomic ratio), and it is still more desirable that it is 90% or more.

[0102] Although Fe occupies the content remainder of an above-mentioned

element, even if it permutes a part of Fe by a kind of Co and nickel, or two sorts of transition-metals elements (T), it can obtain desired hard magnetic property. If the amount of permutations of T to Fe exceeds 50%, the high residual magnetic flux density B_r beyond 0.7T will not be obtained. For this reason, as for the amount of permutations, it is desirable to limit to 50% or less of range 0% or more. In addition, since the Curie temperature of $R_2Fe_{14}B$ phase rises while the square shape nature of a demagnetization curve improves by permuting a part of Fe by Co, thermal resistance improves. The range where the amount of Fe permutations by Co is desirable is 40% or less 0.5% or more.

[0103] Next, the desirable operation gestalt of this invention is explained.

[0104] With a [liquid quenching equipment] book operation gestalt, a raw material alloy is manufactured using the quenching equipment shown in drawing 6, for example. In order to prevent oxidation of the raw material alloy containing the rare earth elements R and Fe which are easy to oxidize, an alloy production process is performed in an inert gas ambient atmosphere. As inert gas, rare gas and nitrogen, such as helium or an argon, can be used. In addition, as for nitrogen, it is desirable to use rare gas, such as helium or an argon, in order to tend to react in comparison with rare earth elements R.

[0105] The equipment of drawing 6 held the vacuum or the inert gas ambient atmosphere, and is equipped with the dissolution room 1 and the quenching room 2 of the raw material alloy which can adjust the pressure. Drawing 6 (a) is a whole block diagram, and drawing 6 (b) is some enlarged drawings.

[0106] As shown in drawing 6 (a), the dissolution room 1 is equipped with the fusion furnace 3 which dissolves the raw material 20 blended so that it might become a desired magnet alloy presentation at an elevated temperature, the hot-water-storing container 4 which has the tapping nozzle 5 at the pars basilaris ossis occipitalis, and the combination feeding equipment 8 for supplying a feed ingredient in a fusion furnace 3, controlling atmospheric penetration. The hot-water-storing container 4 stores the molten metal 21 of a raw material alloy, and has the heating apparatus (un-illustrating) which can maintain the tapping

temperature on predetermined level.

[0107] The quenching room 2 is equipped with the rotation cooling roller 7 for carrying out the rapid solidification of the molten metal 21 which came out of the tapping nozzle 5.

[0108] In this equipment, the ambient atmosphere in the dissolution room 1 and the quenching room 2 and its pressure are controlled by the predetermined range. Therefore, controlled atmosphere feed hopper 1b, 2b and 8b, and the flueing openings 1a, 2a, and 8a are formed in the suitable part of equipment. Especially flueing opening 2a is connected to the pump in order to control the absolute pressure in the quenching room 2 within the limits of 30kPa(s) - ordinary pressure (atmospheric pressure).

[0109] A fusion furnace 3 can be tilted and pours in a molten metal 21 suitably in the hot-water-storing container 4 through a funnel 6. A molten metal 21 is heated by non-illustrated heating apparatus in the hot-water-storing container 4.

[0110] The tapping nozzle 5 of the hot-water-storing container 4 is arranged at the septum of the dissolution room 1 and the quenching room 2, and makes the front face of a cooling roller 7 in which it is located caudad flow down the molten metal 21 in the hot-water-storing container 4. The diameter of an orifice of the tapping nozzle 5 is 0.5-2.0mm. With this operation gestalt, when the viscosity of a molten metal 21 is large, although a molten metal 21 stops being able to flow easily in the inside of the tapping nozzle 5, in order to hold the quenching room 2 in the pressure condition lower than the dissolution room 1, differential pressure is formed between the dissolution room 1 and the quenching room 2, and tapping of a molten metal 21 is performed smoothly.

[0111] A cooling roller 7 may be formed from aluminum alloy from the point of thermal conductivity, a copper alloy, carbon steel, brass, W and Mo, and bronze. However, it is desirable to form from the alloy which contains Cu, Fe or Cu, and Fe from a viewpoint of a mechanical strength and economical efficiency. If a cooling roller is produced with ingredients other than Cu or Fe, since the detachability over the cooling roller of a quenching alloy will worsen, it is [a

possibility that a quenching alloy may coil around a roll] and is not desirable. The diameter of a cooling roller 7 is 300-500mm. The water-cooled capacity of a water cooler established in the cooling roller 7 is computed and adjusted according to the coagulation latent heat and the amount of tapping per unit time amount.

[0112] According to the equipment shown in drawing 6 , the rapid solidification of a total of the 10kg raw material alloy can be carried out, for example in 10 - 20 minutes. In this way, the formed quenching alloy serves as the alloy thin band (thickness:10-300micrometer and width-of-face:2mm-3mm) (alloy ribbon) 22.

[0113] [Melt quenching method] The molten metal 21 of the raw material alloy expressed with the above-mentioned empirical formula is produced first, and it stores in the hot-water-storing container 4 of the dissolution room 1 of drawing 6 . Next, tapping of this molten metal 21 is carried out from the tapping nozzle 5 on the water-cooled roll 7 in a reduced pressure Ar ambient atmosphere, and contact to a cooling roller 7 quenches it, and it is solidified. It is necessary to use the approach of controlling a cooling rate with high precision as the rapid solidification approach.

[0114] In the case of this operation gestalt, it is desirable to carry out a cooling rate on the occasion of the cooling coagulation of a molten metal 21 in 1×10^2 to 1×10^8 degrees C/second, and it is still more desirable to carry out in 1×10^4 to 1×10^6 degrees C/second.

[0115] The time amount by which the molten metal 21 of an alloy is cooled with a cooling roller 7 is equivalent to time amount after an alloy contacts the periphery front face of the rotating cooling roller 7 until it separates, and between them, the temperature of an alloy falls and will be in a supercooled liquid condition. Then, the alloy of a supercooling condition separates from a cooling roller 7, and flies the inside of an inert atmosphere. As a result of heat's being taken by the controlled atmosphere while the alloy is flying with thin band-like one, the temperature falls further. In this invention, since the pressure of a controlled atmosphere is set up within the limits of 30kPa(s) - ordinary pressure, the cooling

effectiveness by the controlled atmosphere can become strong, and a Nd₂Fe₁₄B mold compound can be deposited and grown up uniformly minutely into an alloy. In addition, when the elements M, such as a suitable quantity of Ti, are not added in a raw material alloy, in order that alpha-Fe may deposit and grow preferentially, a final magnet property will deteriorate in the quenching alloy which passed through a cooling process which was mentioned above.

[0116] With this operation gestalt, in order to adjust roll surface velocity a second within the limits of 10m /or more 30m/second or less and to heighten the secondary-cooling-of-concrete effectiveness by the controlled atmosphere, the quenching alloy which contains a detailed R₂Fe₁₄B mold compound phase with a mean particle diameter of 80nm or less more than 60 volume % is produced by setting the ambient-gas-pressure force to 30 or more kPas.

[0117] In addition, the quenching method of an alloy molten metal used by this invention may not be limited to the above-mentioned piece rolling method, but may be the congruence rolling method, the gas atomizing method, the strip cast method that is an approach of not performing control of flow by the nozzle or the orifice, a cooling method which combined the rolling method and the gas atomizing method further.

[0118] Also in the above-mentioned quenching method, the cooling rate of the strip cast method is comparatively low, and is 102-105 degrees C/second. With this operation gestalt, by adding a suitable quantity of Ti into an alloy, even when based on the strip cast method, the quenching alloy with which the organization which does not contain Fe primary phase occupies most can be formed. Since process costs are below one half extent of other melt quenching methods, the strip cast method is effective when producing a lot of quenching alloys compared with the piece rolling method, and is a technique suitable for fertilization. Since the metal texture containing many Fe primary phases generates even if it forms a quenching alloy using the strip cast method when not adding Element M to a raw material alloy, or when Cr, V, Mn, Mo, Ta, and/or W are added instead of Element Ti, a desired metal texture cannot be formed.

[0119] With [heat treatment] book operation gestalt, heat treatment is performed in argon atmosphere. Preferably, a programming rate is carried out in 5 degrees C/second - 20 degrees C/second, and at 550-degree-C or more temperature of 850 degrees C or less, 30 seconds or more, after [20 or less minutes] carrying out time amount maintenance, it cools to a room temperature. Of this heat treatment, into an amorphous phase, the fine crystal of a metastable phase deposits and grows and nano composite organization is formed. According to this invention, it is at the initiation time of heat treatment, and since the detailed Nd₂Fe₁₄B mold crystal phase has already existed more than the whole 60 volume %, big and rough-ization of an alpha-Fe phase or other crystal phases is controlled, and each configuration phase other than a Nd₂Fe₁₄B mold crystal phase (soft magnetism phase) is made detailed by homogeneity.

[0120] In addition, if heat treatment temperature is less than 550 degrees C, many amorphous phases may remain and after heat treatment may not reach level with sufficient coercive force depending on quenching conditions. Moreover, if heat treatment temperature exceeds 850 degrees C, grain growth of each configuration phase will be remarkable, a residual magnetic flux density Br will fall, and the square shape nature of a demagnetization curve will deteriorate. For this reason, although 550 degrees C or more 850 degrees C or less of heat treatment temperature are desirable, the range of more desirable heat treatment temperature is 570 degrees C or more 820 degrees C or less.

[0121] In this invention, it deposits uniformly [the Nd₂Fe₁₄B mold compound phase of amount sufficient in a quenching alloy], and minutely for the secondary-cooling-of-concrete effectiveness by the controlled atmosphere. For this reason, even when not daring perform heat-of-crystallization processing to a quenching alloy, the rapid solidification alloy itself can demonstrate sufficient magnet property. Therefore, although heat-of-crystallization processing is not a process indispensable to this invention, in order for it to be improvement in a magnet property to perform this, it is desirable. In addition, it is possible for heat treatment of low temperature to also fully raise a magnet property as compared

with the former.

[0122] A heat treatment ambient atmosphere has a desirable inert gas ambient atmosphere in order to prevent oxidation of an alloy. 0. You may heat-treat in the vacuum of 1 or less kPa.

[0123] In the quenching alloy before heat treatment, metastable phases, such as Fe₃B phase, Fe₂₃B₆, and an R₂Fe₂₃B three phase circuit, may be contained in addition to the R₂Fe₁₄B mold compound phase and the amorphous phase. In that case, by heat treatment, an R₂Fe₂₃B three phase circuit can disappear, and can carry out crystal growth of the iron machine boride (for example, Fe₂₃B₆) and alpha-Fe which show the saturation magnetization of R₂Fe₁₄B phase, equivalent, or saturation magnetization higher than it.

[0124] Even if a soft magnetism phase like alpha-Fe finally exists in the case of this invention, in order that a soft magnetism phase and a hard magnetism phase may join together magnetically by the exchange interaction, outstanding magnetic properties are demonstrated.

[0125] The diameter of average crystal grain of the R₂Fe₁₄B mold compound phase after heat treatment needs to be set to 300nm or less which is uniaxial crystal particle size, it is desirable that it is [20nm or more] 150nm or less, and it is still more desirable that it is [20nm or more] 100nm or less. On the other hand, if the diameter of average crystal grain of a boride phase or an alpha-Fe phase exceeds 50nm, since the exchange interaction committed to each configuration interphase will become weaker and the square shape nature of a demagnetization curve will deteriorate, (BH) max will fall. If these diameters of average crystal grain are less than 1nm, it becomes impossible to acquire high coercive force. As for the diameter of average crystal grain of soft magnetism phases, such as a boride phase from the above thing, and an alpha-Fe phase, it is desirable that it is [1nm or more] 50nm or less, and it is still more desirable that it is 30nm or less.

[0126] In addition, the thin band of a quenching alloy may be coarsely cut or ground before heat treatment.

[0127] If the obtained magnet is pulverized after heat treatment and magnet powder (magnetic powder) is produced, various bond magnets can be manufactured according to a well-known process from the magnetic powder. When producing a bond magnet, it is mixed with an epoxy resin or Nylon and iron machine rare earth alloy magnetic powder is fabricated by the desired configuration. At this time, other magnetic powder, for example, Sm-Fe-N system magnetic powder, and hard ferrite magnetic powder of a class may be mixed to nano composite magnetic powder.

[0128] Various kinds of rotating machines, such as a motor and an actuator, can be manufactured using an above-mentioned bond magnet.

[0129] When using the ***** end of this invention for injection-molding bond magnets, it is desirable to grind so that average grain size may be set to 200 micrometers or less, and the mean particle diameter of more desirable powder is 30 micrometers or more 150 micrometers or less. Moreover, when using for compression-molding bond magnets, it is desirable to grind so that grain size may be set to 300 micrometers or less, and the mean particle diameter of more desirable powder is 30 micrometers or more 250 micrometers or less. Still more preferably, it has two peaks in particle size distribution, and mean particle diameter is in 50 micrometers or more 200 micrometers or less.

[0130] In addition, regardless of the shaping approach, the moldability at the time of bond magnet shaping, the corrosion resistance of the bond magnet obtained, and thermal resistance are improvable by performing surface preparation, such as coupling processing, chemical conversion, and plating, on the surface of powder. Moreover, as well as powdered surface preparation when surface preparation, such as resin paint, and chemical conversion, plating, is performed to the bond magnet front face after shaping, the corrosion resistance and thermal resistance of a bond magnet can be improved.

[0131]

[Example] First, the presentation ratio x of Q and the presentation ratio z of M explain the example and the example of a comparison with which it is satisfied of

$x < 10 < 15$ atom % and $0.1 < z < 10$ atom %, respectively.

[0132] Weighing capacity was carried out so that a total amount might become 30g using the ingredient of B, C, Fe, Co, Ti, Nd, Pr, Tb, and Dy of 99.5% or more of purity about each of a sample (No.1-No.12) which has the presentation shown in Table 1, and it supplied in the quartz crucible. Here, sample No.1-No.8 are equivalent to the example of this invention, and sample No.9-No.12 are equivalent to the example of a comparison.

[0133]

[Table 1]

| | | 組成 (at%) | | | | 口-A速度 | 融解温度 |
|-------------|----|---------------|-----------|-----------|------|-------|------|
| | | Fe | Q | R | M | m/秒 | ℃ |
| 実 施 例 | 1 | Fe 79 | B 11 | Nd 9 | Ti 1 | 20.0 | 660 |
| | 2 | Fe 78.7 | B 10.3 | Nd 9 | Ti 2 | 12.0 | 700 |
| | 3 | Fe 76.7 | B 10.3 | Nd 9 | Ti 4 | 9.0 | 760 |
| | 4 | Fe 69+Co 3 | B 14 | Nd 3+Pr 3 | Ti 6 | 9.0 | 740 |
| | 5 | Fe 68+Co 3.5 | B 7+C 4 | Nd 9.5 | Ti 8 | 7.0 | 780 |
| | 6 | Fe 78.7 | B 10.3 | Nd 8+Dy 1 | Ti 2 | 12.0 | 720 |
| | 7 | Fe 78.7 | B 5+C 5.3 | Nd 8+Tb 1 | Ti 2 | 12.0 | 720 |
| | 8 | Fe 65.7+Co 10 | B 10.3 | Nd 9 | Ti 6 | 8.0 | 720 |
| 比 較 例 | 9 | Fe 81 | B 12 | Nd 7 | — | 30.0 | 660 |
| | 10 | Fe 80 | B 14 | Nd 6 | — | 20.0 | 680 |
| | 11 | Fe 80.7 | B 10.3 | Nd 9 | — | 25.0 | 660 |
| | 12 | Fe 76.7 | B 10.3 | Nd 11 | Ti 2 | 12.0 | 710 |

[0134] In Table 1, it is shown that "B7+C4" of the column currently displayed as "Q" added B (boron) of 7 atom % and C (carbon) of 4 atom %, and it is shown that "Nd3+Pr3" of the column currently displayed as "R" added Nd of 3 atom % and Pr of 3 atom %.

[0135] Since the quartz crucible used for molten metal production has the orifice with a diameter of 0.8mm at the pars basilaris ossis occipitalis, after dissolving within a quartz crucible, the above-mentioned raw material serves as an alloy molten metal, and will be caudad dropped from an orifice. The pressure performed the dissolution of a raw material using the high-frequency-heating method to the bottom of the argon atmosphere of 1.33kPa(s). In this example, molten metal temperature was set as 1500 degrees C.

[0136] The molten metal was made to blow off to the peripheral face of the

copper roll in the location of 0.7mm of lower parts of an orifice by pressurizing the surface of hot water of an alloy molten metal by Ar gas of 26.7kPa(s). A roll rotates at high speed, while the interior is cooled so that the temperature of the peripheral face may be maintained by room temperature extent. For this reason, the alloy molten metal dropped from the orifice will be flown in the peripheral-velocity direction, contacting a roll peripheral surface and heat being taken. Since an alloy molten metal is continuously dropped on a roll peripheral surface through an orifice, the alloy solidified by quenching will have the gestalt of the ribbon (width of face: 2-3mm, thickness:20-50micrometer) prolonged for a long time in thin band-like one.

[0137] In the case of the roll kneader method (the single rolling method) adopted by this example, a cooling rate is prescribed by roll peripheral velocity and the amount of molten metal flowing down per unit time amount. It depends for this amount of molten metal flowing down on the diameter of an orifice (cross section), and the molten-metal-pressure force. In this example, the diameter of 0.8mm and the molten-metal-pressure force were set to 26.7kPa(s), and the flowing-down rate was set for the orifice as a part for about 0.5-1kg/.

[0138] Roll peripheral velocity was considered as the passage of Table 1.

[0139] Next, the quenching alloy of No.1-No.12 was heat-treated in Ar gas. After specifically holding each quenching alloy for 6 minutes with the heat treatment temperature shown in the rightmost column of Table 1, it cooled to the room temperature. Then, the magnetic properties of each sample were measured using the oscillatory type magnetometer. The following table 2 shows this measurement result.

[0140]

[Table 2]

| | | 磁 気 特 性 | | |
|-------------|----|------------|-----------------|------------------------------------|
| | | B r (T) | H c J (kA/m) | (BH) m a x (kJ/m ³) |
| 実 施 例 | 1 | 0.86 | 490 | 94 |
| | 2 | 0.85 | 605 | 118 |
| | 3 | 0.85 | 695 | 111 |
| | 4 | 0.88 | 520 | 102 |
| | 5 | 0.84 | 740 | 108 |
| | 6 | 0.84 | 658 | 101 |
| | 7 | 0.83 | 682 | 98 |
| | 8 | 0.87 | 730 | 125 |
| 比 較 例 | 9 | 0.80 | 22 | 4 |
| | 10 | 0.69 | 8 | - |
| | 11 | 0.86 | 479 | 80 |
| | 12 | 0.74 | 955 | 88 |

[0141] As shown in Table 2, the magnetic properties of an example were extremely excellent as compared with the magnetic properties of the example of a comparison. Moreover, when Ti was added and the presentation ratio y of rare-earth-elements R (Nd) separated from the range of $6 \leq y < 10$ atom %, the Ti addition effectiveness of the formation of homogeneity detailed of an organization was not fully demonstrated, but the fall of a residual magnetic flux density B_r arose notably.

[0142] Drawing 7 shows the demagnetization curve of the sample (example) of No.2 and No.3, and the sample (example of a comparison) of No.11. The axis of ordinate of the graph of drawing 7 shows magnetization, and the axis of abscissa shows the reinforcement of a demagnetizing field. The square shape nature of the demagnetization curve of an example is very good as compared with the square shape nature of the example of a comparison so that drawing 7 may show. In the case of the example of a comparison, since the diameter of crystal grain is large, it is thought that square shape nature deteriorated.

[0143] Next, the configuration phase was investigated with the characteristic X ray of Cu-Kalpha about each sample of an example. Consequently, in addition to the $R_2Fe_{14}B$ phase, existence of a $Fe_{23}B_6$ phase and an α -Fe phase was checked. On the other hand, in the case of the sample (example of a comparison) of No.9 and No.10, it turned out that the $R_2Fe_{14}B$ mold compound

phase which is hard magnetism is not checked, but the organization which consists of $R_2Fe_{23}B_3$ which is a soft magnetism phase, and α -Fe is formed. Moreover, although the mixed organization which consists of α -Fe which is the $R_2Fe_{14}B$ phase and soft magnetism phase which are a hard magnetism phase was formed by the sample (example of a comparison) of No.11, ferromagnetic iron machine boride was not able to be checked.

[0144] Drawing 8 shows the X diffraction pattern after heat treatment of the sample (example) of No.2 and No.3, and the sample (example of a comparison) of No.11. The axis of ordinate of the graph of drawing 8 shows diffraction reinforcement, and the axis of abscissa shows whenever [angle-of-diffraction].

[0145] In the example, the metal texture which consisted of $Nd_2Fe_{14}B$ phase, an α -Fe phase, and a $Fe_{23}B_6$ phase was formed so that drawing 8 might show. On the other hand, in the example of a comparison, only $Nd_2Fe_{14}B$ phase and an α -Fe phase are observed, but it is thought that B exists superfluously all over an alloy organization.

[0146] In addition, about each sample of No.1-No.8, as a result of investigating the metal texture after heat treatment with a transmission electron microscope, any sample had the nano crystalline structure of 10nm - 25nm or less of diameters of average crystal grain. Moreover, although a part of Ti had permuted by Fe in each configuration phase when the atom probe analyzed the sample of No.2, the great portion of Ti existed in the grain boundary.

[0147] Next, the presentation ratio x of Q and the presentation ratio z of M explain the example with which it is satisfied of $15 \leq x \leq 20$ atom % and $3.0 < z < 12$ atom %, respectively, and the example of reference which is not satisfied.

[0148] Weighing capacity was carried out so that a total amount might become 30g g using the ingredient of B, C, Fe, Co, Ti, and Nd of 99.5% or more of purity about each of a sample (No.13-No.19) which has the presentation shown in Table 3, and it supplied in the quartz crucible.

[0149]

[Table 3]

| | | 組成(at%) | | | | 0-凝固速度 | 熱處理溫度 |
|-----|----|----------------|----------|--------|-------|--------|-------|
| | | Fe | Q | R | M | m/秒 | ℃ |
| 実施例 | 13 | Fe 68.5 | B 15 | Nd 8.5 | Ti 8 | 20 | 680 |
| | 14 | Fe 70.0+Os 2.5 | B 15 | Nd 8.5 | Ti 4 | 20 | 680 |
| | 15 | Fe 71.5 | B 14+C 1 | Nd 8.5 | Ti 5 | 12 | 700 |
| | 16 | Fe 66.5 | B 15 | Nd 8.5 | Ti 10 | 25 | 720 |
| 參考例 | 17 | Fe 76.5 | B 15 | Nd 8.5 | — | 30 | 760 |
| | 18 | Fe 74.5 | B 15 | Nd 8.5 | Ti 2 | 15 | 780 |
| | 19 | Fe 75.5 | B 15 | Nd 6.5 | Ti 3 | 20 | 780 |

[0150] In Table 3, it is shown that "Ti8" of the column currently displayed as "M" added Ti of 8 atom %, and it is shown that the display of "-" has not added Ti.

[0151] The rapid solidification process was performed by the approach same also about sample No.13-19 as the conditions over the above-mentioned sample No.1-12.

[0152] In this way, any sample was an amorphous alloy when the characteristic X ray of CuKalpha investigated the organization of a quenching alloy which got. The reason which was amorphous has comparatively high B concentration in an alloy, and is because it was easy to make it amorphous.

[0153] Next, the quenching alloy of No.12-No.19 was heat-treated in Ar gas. After specifically holding each quenching alloy for 6 minutes with the heat treatment temperature shown in the rightmost column of Table 3, it cooled to the room temperature. Then, the magnetic properties of each sample were measured using the oscillatory type magnetometer. The following table 4 shows this measurement result.

[0154]

[Table 4]

| | 磁 気 特 性 | | |
|----|--------------------|----------------------------|---|
| | B _r (T) | H _c J (kA/m) | (BH) _{max} (kJ/m ³) |
| 13 | 0.83 | 967 | 111 |
| 14 | 0.79 | 908 | 105 |
| 15 | 0.82 | 826 | 104 |
| 16 | 0.70 | 1073 | 78 |
| 17 | 0.63 | 197 | 28 |
| 18 | 0.71 | 462 | 56 |
| 19 | 1.0 | 30 | 12 |

[0155] As shown in Table 4, the magnetic properties of the sample of No.13-16 were excellent as compared with the magnetic properties of the sample (example of reference) of No.17-19.

[0156] Drawing 9 shows the demagnetization curve of the sample of No.13 and No.17. The axis of ordinate of the graph of drawing 9 shows magnetization, and the axis of abscissa shows the reinforcement of a demagnetizing field. The square shape nature of the demagnetization curve of the sample of No.13 is very good as compared with the square shape nature of the sample of No.17 so that drawing 9 may show.

[0157] Drawing 10 and drawing 11 show the X diffraction pattern before and behind heat treatment of the sample of No.13 and No.17, respectively.

[0158] In the case of the example which added Ti, with the alloy before heat treatment (as-spun), the diffraction peak which shows crystallinity is not observed so that drawing 10 may show, but after performing heat treatment for 6 minutes at 660 degrees C, the diffraction peak which shows generation of a compound phase with the Nd₂Fe₁₄B mold crystal structure is observed. That reinforcement is not large although the diffraction peak of an alpha-Fe phase is also observed at this time. When heat treatment temperature is 780 degrees C, the reinforcement of the diffraction peak of an alpha-Fe phase is increasing relatively, and it is presumed that the crystallization temperature of an alpha-Fe phase is higher than the crystallization temperature of Nd₂Fe₁₄B.

[0159] On the other hand, after performing heat treatment for 6 minutes at 600

degrees C as shown in drawing 11 when Ti is not added, the diffraction peak which shows generation of a compound phase with the Nd₂Fe₁₄B mold crystal structure was not observed, but the diffraction peak of an alpha-Fe phase was observed clearly. This shows that the alpha-Fe phase is depositing and growing before crystallization of Nd₂Fe₁₄B phase. When heat treatment temperature is 780 degrees C, the reinforcement of the diffraction peak of an alpha-Fe phase became very strong, and big and rough-ization of an alpha-Fe phase has arisen.

[0160] Thus, when the presentation ratio x of Q is more than 15 atom %, it is desirable to make [more] the presentation ratio z of M than 3.0.

[0161] Next, the molten metal which changed quenching ambient pressure and roll surface velocity was cooled about the alloy which has the presentation of Nd₉Fe_{78.7}B_{10.3}Ti₂ (at%).

[0162] Since the quartz crucible used for molten metal production has the orifice with a diameter of 0.8mm at the pars basilaris ossis occipitalis, after dissolving within a quartz crucible, the above-mentioned raw material serves as an alloy molten metal, and will be caudad dropped from an orifice. The pressure performed the dissolution of a raw material using the high-frequency-heating method to the bottom of the argon atmosphere of 1.33kPa(s). In this example, molten metal temperature was set as 1500 degrees C.

[0163] It was made to flow down a molten metal to the peripheral face of the copper roll in the location of 0.7mm of lower parts of an orifice by pressurizing the surface of hot water of an alloy molten metal by Ar gas of 26.7kPa(s). Other conditions are the same as that of the case of the example mentioned above almost.

[0164] In this example, as shown in the following table 5, the quenching ambient pressure force, roll surface velocity, and heat treatment temperature were changed.

[0165]

[Table 5]

| 試料 | 急冷雰囲気圧 (kPa) | ロール表面速度 (m/秒) | 熱処理温度 (°C) |
|----|-----------------|------------------|---------------|
| 20 | 40.0 | 10.0 | 620 |
| 21 | 35.0 | 15.0 | 640 |
| 22 | 40.0 | 20.0 | 650 |
| 23 | 80.0 | 23.0 | 660 |
| 24 | 60.0 | 12.0 | 640 |
| 25 | 40.0 | 28.0 | 690 |
| 26 | 10.0 | 15.0 | 680 |
| 27 | 40.0 | 35.0 | 700 |
| 28 | 40.0 | 5.0 | 800 |

[0166] The characteristic X ray of Cu-K α investigated the organization of the quenching alloy produced by the above-mentioned melt quenching method. By the sample of No.20-25, the Nd₂Fe₁₄ B phase all checked being contained more than the whole 60 volume % by TEM (transmission electron microscope).

Moreover, an α -Fe phase and existence of Fe₂₃B₃ were also observed in addition to the Nd₂Fe₁₄ B phase. Drawing 12 shows the X diffraction pattern of the sample of No.21. The profile indicated to be "as-spun" is the X diffraction pattern of the quenching alloy before heat-of-crystallization processing among drawing 12 . Moreover, the X diffraction pattern after the heat-of-crystallization processing mentioned later is also shown in drawing 12 .

[0167] Although the diffraction peak by Nd₂Fe₁₄ B phase, the α -Fe phase, and the Fe₂₃ B₆ phase was checked by the sample of No.26, by the sample of No.27, only the halo pattern was observed and the strong diffraction peak by the α -Fe phase and few diffraction peaks by Nd₂Fe₁₄ B phase were observed in sample No.28. In addition, in the case of sample No.26, many amorphous phases existed.

[0168] Next, the quenching alloy of No.20-No.26 was heat-treated in Ar gas. After specifically holding each quenching alloy for 6 minutes with the heat treatment temperature shown in the rightmost column of the above-mentioned table 5, it cooled to the room temperature. Then, the magnetic properties of each sample were measured using the oscillatory type magnetometer. The following table 6 shows this measurement result.

[0169]

[Table 6]

| 試料 | 磁 気 特 性 | | |
|----|--------------------|------------------------|--|
| | B _r (T) | H _{cJ} (kA/m) | (BH) _{max} (kJ/m ³) |
| 20 | 0.89 | 705 | 124 |
| 21 | 0.94 | 650 | 130 |
| 22 | 0.95 | 600 | 126 |
| 23 | 0.88 | 683 | 124 |
| 24 | 0.90 | 670 | 125 |
| 25 | 0.87 | 588 | 120 |
| 26 | 0.83 | 780 | 114 |
| 27 | 0.81 | 754 | 103 |
| 28 | 0.64 | 334 | 38 |

[0170] As shown in Table 6, by the sample of No.20-25, the hard magnetic property which was excellent in residual magnetic flux density $B_r \geq 0.85T$, proper coercive force $H_{cJ} \geq 480$ kA/m, and maximum energy product $(BH)_{max} \geq 120$ kJ/m³ was obtained.

[0171] Drawing 13 shows the demagnetization curve of the sample of No.21, and the sample of No.26. The axis of ordinate of the graph of drawing 13 shows magnetization, and the axis of abscissa shows the reinforcement of a demagnetizing field. The square shape nature of the demagnetization curve of the sample of No.21 is very good as compared with the square shape nature of the sample of No.26 so that drawing 13 may show. In the case of the sample of No.26, since the diameter of crystal grain is large, it is thought that square shape nature has deteriorated.

[0172] Next, about each sample of the example after heat treatment, the configuration phase was investigated using the characteristic X ray of Cu-Kalpha, and crystal size was measured using TEM. Consequently, the diameter of average crystal grain of R2Fe14 B phase was in the range of 20-100nm, and the diameter of average crystal grain of an alpha-Fe phase and an iron machine boride phase was in the range of 10-50nm.

[0173] On the other hand, in the case of the sample of No.26 and No.28, the difference was not produced in the class of configuration phase before and after

heat treatment, but in the case of sample No.27, in addition to the R2Fe14B mold compound phase, a deposit and growth of an alpha-Fe phase and Fe23 B6 were checked.

[0174] It is desirable to set quenching ambient pressure to 30 or more kPas from the above thing, and when setting a quenching ambient atmosphere to 30 or more kPas, it is desirable to carry out [second] roll surface velocity a second in 10m /or more 30m /or less.

[0175]

[Effect of the Invention] According to this invention, the permanent magnet which demonstrates the magnetic properties in which coercive force and magnetization were enough highly excellent is obtained by performing quenching of the alloy molten metal which added Ti, reducing the amount of rare earth elements required for a magnet.

[0176] Moreover, in case a quenching alloy is produced by adding Ti using a melt quenching method according to this invention, even if it reduces a cooling rate, the deposit of the alpha-Fe phase at the time of a liquid cooling process is controlled. Therefore, a cooling rate is comparatively slow like the strip cast method, and since it becomes possible to use the melt quenching method suitable for fertilization, it is very effective in reduction of a manufacturing cost.

[Translation done.]

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] It is the graph which shows the relation of the maximum MAG energy-product (BH) max and boron concentration of the Nd-Fe-B nano composite magnet with which Ti is not added. A white bar shows the data of the sample containing 10 - 14at% Nd among a graph, and the black bar shows the data of the sample containing 8 - 10at% Nd.

[Drawing 2] It is the graph which shows the relation of the maximum MAG energy-product (BH) max and boron concentration of the Nd-Fe-B nano composite magnet with which Ti was added. A white bar shows the data of the sample containing 10 - 14at% Nd among a graph, and the black bar shows the data of the sample containing 8 - 10at% Nd.

[Drawing 3] It is drawing showing the secondary-cooling-of-concrete effectiveness by the atmospheric-air controlled atmosphere. An axis of abscissa shows the elapsed time (Time) from cooling process initiation, and the axis of ordinate shows the temperature (Temperature) of the alloy currently cooled.

[Drawing 4] It is the mimetic diagram showing the $R_2Fe_{14}B$ mold compound phase and (Fe, Ti)-B phase in a magnet by this invention.

[Drawing 5] When Ti is added, it is drawing showing typically the change of a detailed organization in the crystallization process of the rapid solidification alloy at the time of replacing with Ti and adding Nb etc.

[Drawing 6] (a) is the sectional view showing the whole equipment example of a configuration used for the approach of manufacturing the quenching alloy for the iron machine rare earth alloy magnet by this invention, and (b) is an enlarged drawing of a part with which rapid solidification is performed.

[Drawing 7] It is the graph which shows the demagnetization curve of the sample (example) of No.2 and No.3, and the sample (example of a comparison) of No.11.

[Drawing 8] It is the graph which shows the X diffraction pattern after heat

treatment of the sample of No.2 and No.3, and the sample of No.11.

[Drawing 9] It is the graph which shows the demagnetization curve of the sample of No.14 and No.19.

[Drawing 10] It is the graph which shows the X diffraction pattern before and behind heat treatment of No.14.

[Drawing 11] It is the graph which shows the X diffraction pattern before and behind heat treatment of the sample of No.19.

[Drawing 12] It is the graph which shows the X diffraction pattern in the quenching alloy (before heat-of-crystallization processing) of the sample of No.21, and the X diffraction pattern after heat-of-crystallization processing (640-degree-C 6 minutes). An axis of ordinate is the reinforcement (Intensity) of a diffraction peak, and an axis of abscissa is whenever [angle-of-diffraction].

[Drawing 13] It is the graph which shows the demagnetization curve of the sample of No.21, and the sample of No.26.

[Description of Notations]

1b, 2b, 8b and 9b Controlled atmosphere feed hopper

1a, 2a, 8a, and 9a Flueing opening

1 Dissolution Room

2 Quenching Room

3 Fusion Furnace

4 Hot-Water-Storing Container

5 Tapping Nozzle

6 Funnel

7 Rotation Cooling Roller

21 Molten Metal

22 Alloy Thin Band

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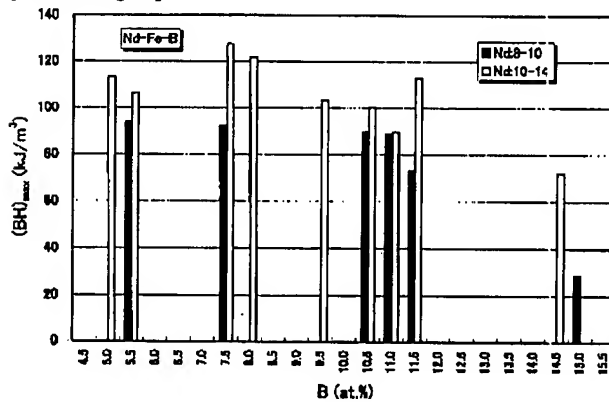
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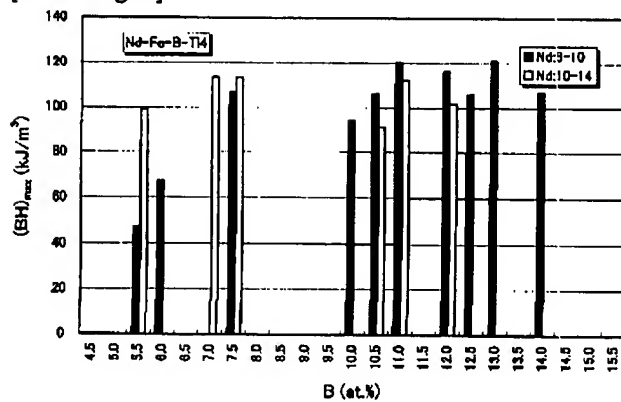
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DRAWINGS

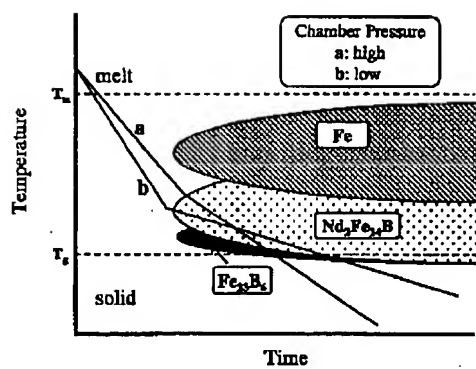
[Drawing 1]



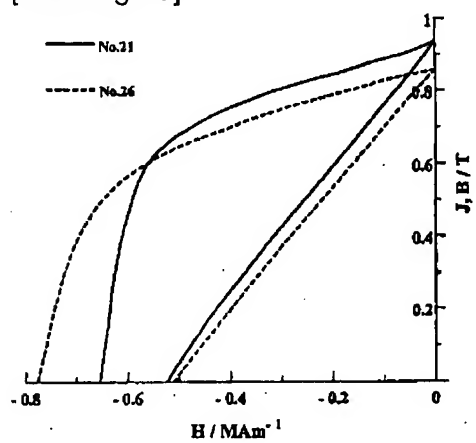
[Drawing 2]



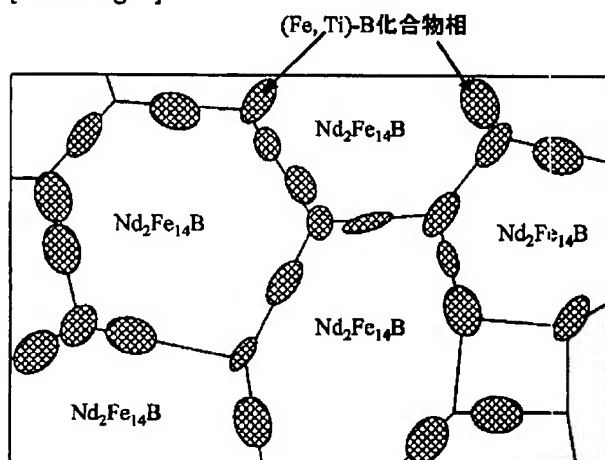
[Drawing 3]



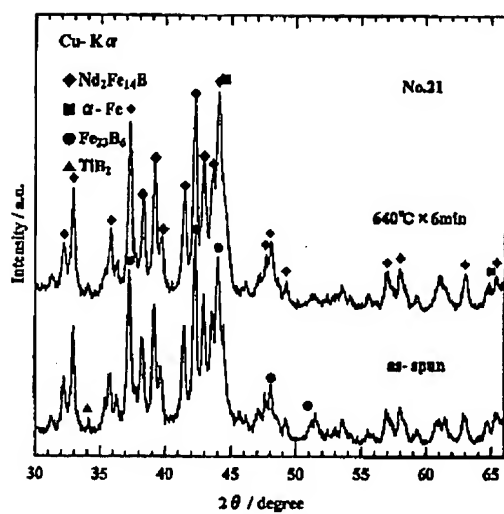
[Drawing 13]



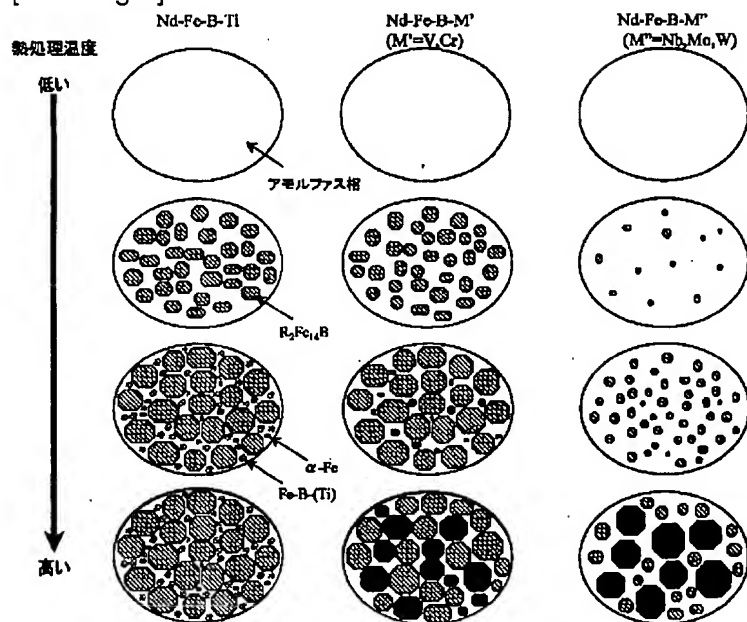
[Drawing 4]



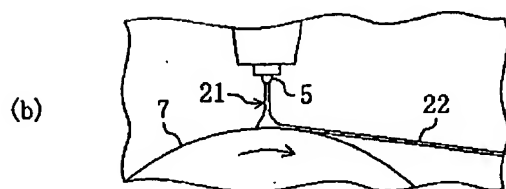
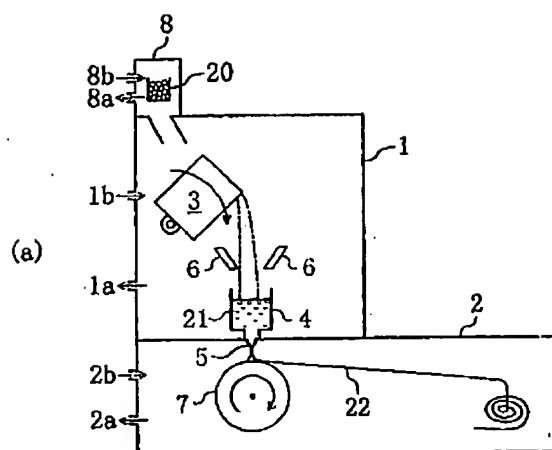
[Drawing 12]



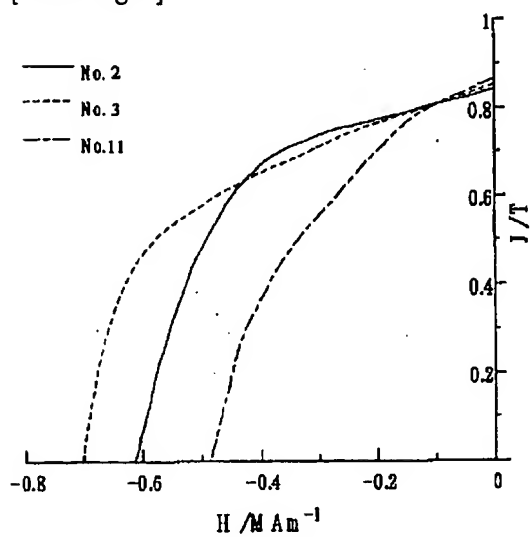
[Drawing 5]



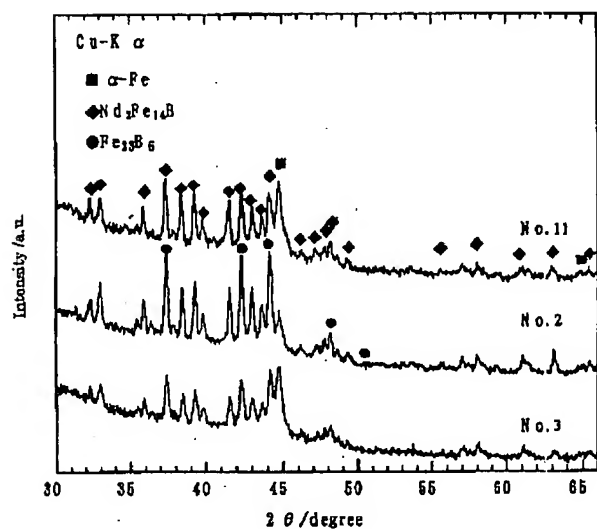
[Drawing 6]



[Drawing 7]

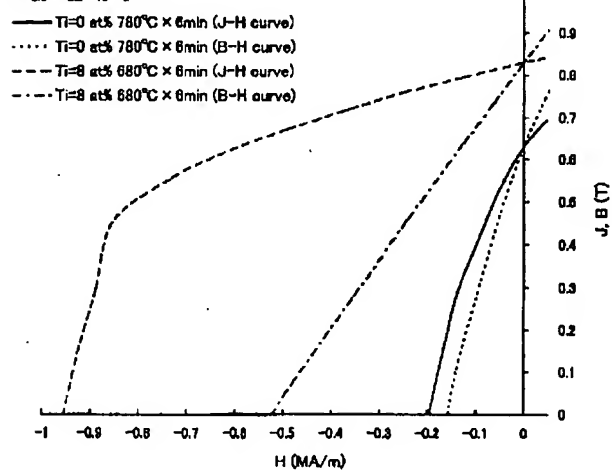


[Drawing 8]



[Drawing 9]

$\text{Nd}_{0.5}\text{Fe}_{0.5}\text{B}_{15}\text{Ti}_2$ at%



[Drawing 10]